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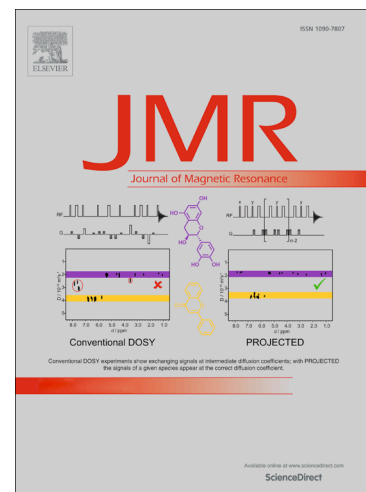
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# Setting the magic angle for fast magic-angle spinning probes

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## Abstract

Fast magic-angle spinning, coupled with  $^1\text{H}$  detection is a powerful method to improve spectral resolution and signal to noise in solid-state NMR spectra. Commercial probes now provide spinning frequencies in excess of 100 kHz. Then, one has sufficient resolution in the  $^1\text{H}$  dimension to directly detect protons, which have a gyromagnetic ratio approximately four times larger than  $^{13}\text{C}$  spins. However, the gains in sensitivity can quickly be lost if the rotation angle is not set precisely. The most common method of magic-angle calibration is to optimize the number of rotary echoes, or sideband intensity, observed on a sample of KBr. However, this typically uses relatively low spinning frequencies, where the spinning of fast-MAS probes is often unstable, and detection on the  $^{13}\text{C}$  channel, for which fast-MAS probes are typically not optimized. Therefore, we compare the KBr-based optimization of the magic angle with two alternative approaches: optimization of the splitting observed in  $^{13}\text{C}$ -labeled glycine-ethylester on the carbonyl due to the  $\text{C}\alpha\text{-C}'$   $J$ -coupling, or optimization of the  $\text{H-N}$   $J$ -coupling spin echo in the protein sample itself. The latter method has the particular advantage that no separate sample is necessary for the magic-angle optimization.

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